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Registry No. $[2((CH_3)_2N)_2PC]$ ·AlCl₃, 60594-85-6; $((CH_3)_2-N)_3P$ · $((CH_3)_2N)_2PC]$ ·AlCl₃, 60594-83-4; $((CH_3)_2N)_3P$ ·AlCl₃, 60594-91-4; ³¹P, 7723-14-0.

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Preparation, Structure, and Properties of Ytterbium Ruthenium Hydride

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Yb₂RuH₆, a black polycrystalline solid, was formed by heating YbH₂ and ruthenium powders at 800 °C in approximately 1 atm of hydrogen. X-ray powder diffraction data conformed to the face-centered cubic Bravais system, space group *Fm3m*, with a = 7.248 Å. The structure was consistent with the metals arranged in a fluorite type lattice and in such a way that the ruthenium and ytterbium ions occupied the eight- and fourfold sites, respectively. The gram-susceptibility varied from about 3×10^{-6} emu/g at room temperature to about 8×10^{-6} emu/g at liquid N₂ temperatures and departed from Curie–Weiss behavior in this region. Arguments are advanced showing that ytterbium and ruthenium in the ternary hydride are divalent. The electrical resistivity of Yb₂RuH₆ was consistent with semiconductor behavior with $\Delta E = 0.18$ eV.

Introduction

Several investigations have been published drawing attention to the formation of some interesting ternary hydrides prepared by exposing binary alloys composed of lanthanum or a rare earth element and cobalt or nickel to hydrogen at or slightly above room temperature and at pressures above 1 atm. A particularly attractive feature of these alloys is their capacity to absorb large quantities of hydrogen. For example, LaNi₅H_{6.7} is formed by exposing LaNi₅ to 2.5 atm of hydrogen at room temperature, and SmCo₅H₃, by the roomtemperature hydrogen absorption of SmCo₅ at 4.5 atm.¹ DyCo₃H₅, HoCo₃H₅, and ErCo₃H₅ are formed at room temperature by exposing the respective rare earth-cobalt alloys to hydrogen at 40 atm.² Our group, which is studying the formation, structure, and properties of rare earth-rarer platinum metal ternary hydrides, reported recently the formation of Eu₂RuH₆.³ We wish to report now the results of our investigation of Yb₂RuH₆.⁴

Experimental Section

Ytterbium ingot, presumably 99.9% pure, was purchased from A. D. MacKay, Inc., and from Electronic Space Products, Inc. The metal was purified further by vacuum distillation at 800 °C. In order to reduce the possibility of ferromagnetic contamination from the stainless steel distillation cold finger, the ytterbium was collected on a molybdenum disk, 0.005 in. thick, located at the tip of the cold finger. Ruthenium powder, 99.9% pure and 325 mesh, was purchased from A. D. MacKay, Inc. Prepurified grade hydrogen, purchased from

*To whom correspondence should be addressed: R. Lindsay, Department of Physics; R. O. Moyer, Jr., Department of Chemistry Airco Industries, Inc., was purified further by passing the gas through a hot palladium tube filter, Model A-1-DH, purchased from Matthey Bishop, Inc. Prepurified argon (99.998% minimum) and prepurified nitrogen (99.995%) were purchased from Airco Industrial Gases, Inc.

Distilled ytterbium was cut into chips, approximately 1/4 in. on edge and placed into a molybdenum boat, which in turn was loaded into a quartz reaction tube. The tube was attached to a glass vacuum line, and the system was evacuated to at least 10^{-5} mm of mercury. Hydrogen was introduced into the system to approximately 700 mm of mercury, and the ytterbium was heated to 500 °C. The temperature was maintained at 500 °C for 4–5 h. Anal. Calcd for YbH₂: Yb, 98.85; H, 1.15. Found: Yb, 97.71; H, 1.12. X-ray powder diffraction data were consistent with the data published for ytterbium deuteride.⁵

Ytterbium ruthenium hydride was prepared by thoroughly mixing ytterbium hydride and ruthenium powders in a 2:1 mole ratio, respectively. A powdered mixture containing 0.7375 g of YbH₂ and 0.2131 g of ruthenium was typical of the reactant size and was compressed at 5000 psi into a 1/4-in. diameter pellet. The procedure for the high-temperature synthesis was similar to that reported elsewhere for Eu₂RuH₆.³ The reactant pellet was heated at 800 °C for approximately 18 h in hydrogen at approximately 700 mm of mercury.

Elemental analyses of ytterbium and ruthenium were performed by gravimetric methods. Hydrogen was determined by thermal decomposition of the sample and its subsequent transfer to a calibrated gas buret. Procedures for the analyses are given elsewhere.³

Procedures for the x-ray diffraction, magnetic susceptibility, density, and electrical resistivity measurements have been reported elsewhere.³ Deviations from these procedures are given below. X-ray powder diffraction intensities were determined by cutting the tracings above the background and weighing the paper on an analytical balance. Electrical resistivities were obtained from measurements on a pellet

Table I. X-Ray Powder Diffraction Data for Yb, RuH₆

Sin ² θ		Sin ² θ				Sin ² θ			
	hkl	Obsd	Calcd	hkl	Obsd	Calcd	hkl	Obsd	Calcd
	111	0.0344	0.0339	422	0.2710	0.2714	551,711	0.5768	0.5768
	200	0.0458	0.0452	333, 511	0.3045	0.3053	640	0.5885	0.5881
	220	0.0907	0.0905	440	0.3624	0.3619	642	0.6321	0.6333
	311	0.1243	0.1244	531	0.3963	0.3958	731.553	0.6668	0.6672
	222	0.1356	0.1357	600, 442	0.4066	0.4071	644	0.7688	0.7690
	400	0.1809	0.1809	620	0.4529	0.4524	660, 822	0.8136	0.8143
	331	0.2148	0.2149	622	0.4975	0.4976	840	0.9044	0.9047
	420	0.2264	0.2264	444	0.5435	0.5428	842	0.9494	0.9500

with a length and area of 0.340 cm and 0.336 cm², respectively. Electrical resistances were measured at 293, 200, 145, and 77 K.

Results and Discussion

Ytterbium ruthenium hydride is formed by heating ytterbium hydride and ruthenium powders at 800 °C in approximately 1 atm of hydrogen. During the formation of the ternary hydride, the reactant pellet expanded substantially, and the hydrogen pressure in the system decreased. The synthesis turned out to be complicated by a considerable weight loss of the reactants. For example, starting with a 2:1 mole ratio of ytterbium hydride to ruthenium, heated at 800 °C for approximately 18 h, the sample lost 20% of its weight, presumably due to the evaporation of YbH_2 . It is believed that the YbH₂ volatilized and reacted directly with the quartz sleeve surrounding the sample. The increase in the weight of the quartz sleeve was consistent with the weight loss of the sample within 1%. X-ray powder diffraction data indicated that the product was composed of the ternary hydride single phase and elemental ruthenium. Starting with a 2:1 mole ratio of YbH_2 to Ru, a 20% weight loss corresponded to a product contaminated with approximately 6% ruthenium. Anal. Calcd for Yb₂RuH₆: Yb, 76.37; Ru, 22.30; H, 1.33. Found: Yb, 75.77; Ru, 21.83; H, 1.22. The following equation represents the synthesis of Yb₂RuH₆

 $2YbH_2(s) + Ru(s) + H_2(g) \rightarrow Yb_2RuH_6(s)$

 Yb_2RuH_6 , like YbH_2 , is a black polycrystalline solid which is reactive to acidic solutions.

Haschke and Clark⁶ studied the vaporization of YbH₂ and reported that between 726 and 930 K, YbH₂(s) vaporized according to the equation

 $YbH_2(s) \rightarrow Yb(g) + H_2(g)$

Using their data, it appears that at 930 K the vapor pressure of ytterbium in equilibrium with H_2 and $YbH_2(s)$ approaches 10^{-3} atm.

Attempts to compensate for the loss of YbH₂ were made. For example, a 0.5 molar excess of YbH₂ was added to a 2:1 mole ratio mixture to compensate for the YbH₂ loss during the course of the synthesis. The reactants were heated at 800 °C for approximately 18 h in approximately 1 atm of hydrogen. The shift in the mole ratio of ytterbium hydride to ruthenium of 2:1 to 2.5:1 did not yield the desired result. The x-ray diffraction pattern of the product revealed unreacted YbH₂ as well as Yb₂RuH₆. An attempt to mitigate the rate of YbH₂ loss by reducing the reaction temperature to 750 °C resulted in an incomplete reaction. The x-ray powder diffraction pattern of the product revealed unreacted YbH₂ and Ru as well as the ternary hydride. Unfortunately, equipment was not accessible to determine whether the YbH₂ loss could be reduced by increasing the hydrogen pressure above 1 atm.

The x-ray powder diffraction data of the ternary hydride phase were indexed on the basis of face-centered cubic symmetry. The results of this analysis are given in Table I.

An extrapolation of the measured lattice parameters to $\sin^2 \theta = 1$ by reducing the data with a least-squares analysis technique gave a unit cell dimension of 7.248 Å.

Table II. Observed and Calculated X-Ray Diffraction Intensities for Yb_2RuH_6

hkl	Ι _o	Ic	hkl	Io	I _c	
 111	15.42	13.58	422	32.90	33.82	
200	29.16	36.71	511, 333	2.94	1.84	
220	100	100	440	8.24	10.06	
311	8.13	3.35	600, 442	3.90	6.42	
222	10.75	11.11	620	11.28	13.84	
400	18.32	17.33	622	12.63	20.78	
331	2.52	2.65	444	1.32	3.68	
420	9.52	14.09	640	2.30	3.61	
			642	1.31	3.07	

The experimental density of Yb_2RuH_6 after correcting for the ruthenium contamination was 7.68 g/cm³. This value compares favorably with four formula units of Yb_2RuH_6 having an x-ray density of 7.86 g/cm³.

The structure factor analysis of the x-ray powder diffraction data revealed that Yb₂RuH₆ belonged to the *Fm*3*m* space group. The most successful trial structure can be viewed in terms of the fluorite lattice wherein the ytterbium ions occupy the fourfold sites with atomic coordinates at $(^{1}/_{4}, ^{1}/_{4}, ^{1}/_{4})$, $(^{3}/_{4}, ^{1}/_{4}, ^{1}/_{4}), (^{1}/_{4}, ^{3}/_{4}, ^{1}/_{4}), (^{3}/_{4}, ^{3}/_{4}, ^{3}/_{4}), (^{1}/_{4}, ^{1}/_{4}, ^{3}/_{4}),$ $(^{3}/_{4}, ^{1}/_{4}, ^{1}/_{4}), (^{3}/_{4}, ^{3}/_{4}, ^{3}/_{4}), (^{1}/_{4}, ^{1}/_{4}, ^{3}/_{4}),$ $(^{3}/_{4}, ^{1}/_{4}, ^{3}/_{4}), (^{3}/_{4}, ^{3}/_{4}, ^{1}/_{4}),$ and $(^{1}/_{4}, ^{3}/_{4}, ^{3}/_{4})$; and the europium ions occupy the eightfold sites at (0, 0, 0), $(^{1}/_{2}, ^{1}/_{2},$ $0), (^{1}/_{2}, 0, ^{1}/_{2})$, and $(0, ^{1}/_{2}, ^{1}/_{2})$. Hydrogen ions were not inserted into the trial structures because of their low scattering capacity for x rays. The calculated intensities are shown in Table II wherein the data have been temperature corrected. The reliability factor for the data in Table II, defined as R= $\sum |I_0 - I_c| / \sum I_0$, is 0.15.

Yb₂RuH₆ appears to be isostructural with Eu₂RuH₆³ and Sr₂RuH₆.⁷ A neutron diffraction study⁷ for Sr₂RuD₆ revealed that ruthenium is six-coordinate with respect to deuterium, wherein the deuteriums occupy sites at (0, x, 0) with x approximately equal to 0.25.

The magnetic susceptibility revealed a very small field dependence over the range 3000-7900 Oe implying a ferromagnetic impurity. The ferromagnetic source in this particular sample may be due to a small contamination by the stainless steel cold finger during the distillation of ytterbium because it was observed that the magnetic susceptibility of the YbH_2 used to prepare Yb₂RuH₆ displayed a small field dependence also. However, it may also have resulted from some contamination of the original ytterbium metal since its susceptibility measurements showed small field dependences. The measured magnetic moment of the impurity could have been produced by as little as 0.001% by weight of a ferromagnetic metal such as iron. The magnetic susceptibility of the ruthenium metal powder was measured and the results showed an essentially field- and temperature-independent susceptibility, i.e., 0.44×10^{-6} and 0.52×10^{-6} emu/g at 77 and 296 K, respectively. The room-temperature susceptibility was consistent with the reported values for ruthenium.⁸ Since the gram-susceptibility of the Yb₂RuH₆ was linear with respect to the reciprocal of the magnetic field, the susceptibility at each temperature under conditions of field saturation could be determined by extrapolating the magnetic susceptibility to

Table III.	Magnetic	Susceptibility	v vs. Tem	perature for	Yb.RuH.

	<i>Т</i> , К	$10^6 \chi_{g}$, cgsu	$10^3 \chi_{\rm m}$, cg su	$1/\chi_{\rm m}$, cgsu ⁻¹
	284 264 244 226 198 175 150 136 123 114 105 93 80	3.40 3.48 3.65 3.87 4.11 4.66 5.13 5.61 6.07 6.41 6.82 7.63 8.72	1.61 1.64 1.72 1.82 1.93 2.18 2.39 2.61 2.82 2.97 3.16 3.53 4.02	622 609 581 549 518 459 418 383 355 336 317 284 249
l/y _M (emu/mole)-1	700 600 500 400 300 200 100		20 ⁰ 00	5000
		100 Te	200 mperatu r e ('	300 РК)

Figure 1. Graph of χ_m^{-1} [(emu/mol)⁻¹] vs. T (K) for Yb₂RuH₆.

infinite field strength. Furthermore, the susceptibility was corrected for the ruthenium contamination and the diamagnetism of the ion cores.9 Table III gives the final corrected gram-susceptibilities (χ_g) and molar susceptibilities (χ_m) at infinite field over a temperature range of 80-284 K. The reciprocal of the molar susceptibility for Yb₂RuH₆ is plotted vs. the absolute temperature in Figure 1.

In an attempt to establish the oxidation states of the ytterbium and ruthenium on the basis of the magnetic susceptibility data, three models are possible: $Yb^{II}_2Ru^{II}H_6$, Yb^{III}₂Ru⁶H₆, and Yb^{III}Yb^{II}Ru^IH₆. It is assumed that for each model the ruthenium ions are six-coordinate with respect to hydrogen and that the hybridized d²sp³ orbitals of the ruthenium are energetically separated so as to give a low-sin configuration.

We start by considering what the theoretical effective magnetic moments in Bohr magnetons per formula unit should be for each of these models. The moment for $Yb^{II}_2Ru^{II}H_6$ should be zero for the following reasons. The Yb²⁺ moment is zero because of a ground term with zero orbital and spin quantum numbers while the Ru²⁺ moment is zero because the six 4d electrons occupy completely the three t_{2g} orbitals. In the Yb^{III}₂Ru⁰H₆ model each Yb³⁺ ion contributes a moment of 4.5 $\mu_{\rm B}$,¹⁰ based on a ground term of ²F with an orbital quantum number of L = 3 and a spin quantum number of S = 1/2. If it is assumed that Ru⁰ has eight 4d electrons, six of which occupy completely the t_{2g} orbitals and two of which are located in eg orbitals, the effective moment based on a spin only calculation equals 2.83 μ B. These will add up to a total effective formula unit moment of 11.8 μ_B . The ruthenium in the mixed-valence Yb^{III}Yb^{II}Ru^IH₆ model should contribute a moment consistent with a spin-only value of 1.73 μ_B arising from the single-electron occupancy in an e_g orbital, which would then add to the moment of the Yb³⁺ ion to give a total of 6.2 μ_B per formula unit. When these values are compared with the observed results on Yb_2RuH_6 , it is clear there is no

Tal

ble Iv. Magne	tic Susception	ity vs. Tempera	ture for YDH ₂
<i>Т</i> , К	χ_{g} , cgsu	10⁴x _m , cgsu	$1/\chi_{\mathbf{m}}$, cgsu ⁻¹
274	3.15	5.17	1750
26 0	3.26	5.91	1690
231	3.49	6.31	1580
215	3.62	6.54	1530
199	3.84	6.92	1445
184	4.11	7.39	1352
170	4.32	7.76	1288
154	4.56	8.18	1222
140	5.01	8.97	1115
125	5.38	9.62	1040
109	5.88	10.5	953
98	6.51	11.6	862
87	7.13	12.7	789
7 9	7.60	13.5	741
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Figure 2. Graphs of χ_m^{-1} [(emu/mol)⁻¹] vs. T (K) for YbH₂ and χ_A^{-1} [(emu/g-atom)⁻¹] vs. T (K) for Yb₂O₃.

neat fit for any of the models.

It will be noted that $1/\chi_m$ is not linear in T implying that the Curie-Weiss law is not obeyed over the complete range of temperatures used. For purposes of rough comparison and for reasons we will cite later we will assume the high-temperature slope is more closely identified with Curie-Weiss behavior. Between 175 and 284 K the curve is representative of approximately 2.2 μ_B per formula weight.

These values fall closer to the values of $Yb^{II}_2Ru^{II}H_6$ than of either of the other two but the sizable difference still prompted us to carry out further investigations in an attempt to clarify the result.

We measured the magnetic susceptibility of YbH₂ from 79 to 274 K. Like the Yb_2RuH_6 sample, this also exhibited a small field-dependent paramagnetic susceptibility. The corrected gram-susceptibilities and molar susceptibilities are given in Table IV. Figure 2 is a plot of the inverse of the molar susceptibility vs. the absolute temperature for YbH₂. Like the previous curve for Yb_2RuH_6 in Figure 1 it does not follow the Curie-Weiss law.

Ideally, YbH₂ should be diamagnetic, so that the small observed paramagnetism was unexpected. Wallace, Kubota, and Zanowick¹¹ reported that the gram-susceptibilities for ytterbium and YbH₂ were of the order of 10^{-6} emu/g with YbH₂ being more weakly paramagnetic than ytterbium. Furthermore, Warf and Hartcastle¹² reported on the roomtemperature gram-susceptibility for YbH2 and observed that the susceptibility appeared to increase with an increase in the ratio of hydrogen to ytterbium. For example, as H:Yb shifted from 1.88 to 2.04, the gram-susceptibility increased from 1.50 \times 10⁻⁶ to 5.06 \times 10⁻⁶ emu/g. This paramagnetism was

Table V. Electrical Resistivity for Yb₂RuH₆

Т, К	ρ, Ω cm	Т, К	ρ, Ω cm
293	3.06×10^{4}	145	1.09 × 10 ⁶
200	$2.27 imes 10^{5}$	77	6.52×10^{8}

interpreted in terms of a small proportion of the divalent ytterbium losing electrons and becoming trivalent, with the electrons entering the conduction band.

There was also the possibility that the source of paramagnetism might be due to a small quantity of Yb₂O₃ impurity. Hacker, Lin, and Westrum¹³ have reported the magnetic susceptibility for Yb₂O₃ and showed that the temperature dependence of the susceptibility did not conform to the Curie-Weiss law between 4 and 300 K. Their data¹³ in terms of the reciprocal of the molar susceptibility vs. the absolute temperature are given in Figure 2. A visual comparison of the curves for the inverse of the susceptibility vs. the absolute temperature in Figures 1 and 2 for Yb₂RuH₆, YbH₂, and Yb₂O₃ shows a strong similarity in form with all three being concave toward the T axis.

An investigation was undertaken to ascertain the magnetic susceptibility of reactants and products at different stages in the formation of Yb_2RuH_6 . The source of the ytterbium used to form YbH₂ and then Yb₂RuH₆ was followed from the initial chip of unrefined metal as received from the supplier. It was found that the ytterbium metal showed a distinct drop in susceptibility after being vacuum-distilled. This was interpreted as being due to the elimination of contaminants, particularly ytterbium oxide. A chip of YbH₂ which had not been mechanically handled in any way was then studied and found to have an appreciably lower susceptibility (0.71×10^{-6}) emu/g) at room temperature than powdered YbH₂ which had been formed by crushing YbH₂ chips in an agate vial under a nitrogen or argon atmosphere $(2.38 \times 10^{-6} \text{ emu/g})$. The fact that, at best, the glovebag is not completely "airtight" and that the surface area of YbH2 is increased substantially during crushing may create conditions favorable for the formation of a small amount of ytterbium oxide which would ultimately cause the gram-susceptibility of the YbH₂ to increase. Elemental analyses of a typical powdered sample of YbH₂ showed 97.71 wt % Yb and 1.12 wt. % H. If it is assumed that the weight percentage difference of 1.17 is due entirely to oxygen, then it might be possible for the sample to have as much as 9.6% Yb₂O₃. If the susceptibility data of Hacker et al.¹³ on Yb₂O₃ are applied, the difference between the susceptibilities of the powdered YbH_2 and chips of YbH_2 can be accounted for. The apparent departure from Curie-Weiss behavior of the YbH₂ shown in Figure 2 can then also be attributed to the influence of the oxide. Hacker et al.¹³ have observed this deviation effect in the oxide between 4 and 300 K and attributed it to a splitting of the ground state into four levels with a temperature-dependent occupancy which makes the effective moment a function of temperature.

The order of magnitude of the susceptibility of the uncrushed YbH₂ is too small to be accounted for by anything but ytterbium in the divalent state. Like europium, but in contrast to the other rare earth metals, ytterbium appears to resist the formation of a trivalent binary hydride at 1 atm of hydrogen. Warf and Hardcastle¹⁴ have shown that a higher hydride of ytterbium, $YbH_{2.57}$, is found only after the hydrogen pressure is raised to 22 atm.

Because the synthesis of Yb₂RuH₆ by our technique requires powdered YbH₂ as starting material, the magnitude of the susceptibility for Yb₂RuH₆ and its apparent nonconformity to Curie-Weiss behavior can be explained as due to the influence of ytterbium oxide. Since the magnitude of the atomic gram-susceptibility of Yb₂RuH₆ differed little from that of YbH_2 , we conclude there is no change in the oxidation state of ytterbium in the formation of Yb₂RuH₆ from YbH₂.



Figure 3. Graph of $\log \rho$ (Ω cm) vs. T (K) for Yb₂RuH₆.

Therefore, the most appropriate model for Yb₂RuH₆, like Eu₂RuH₆, is that with ytterbium in the divalent state as represented by Yb^{III}₂Ru^{II}H₆.

The results of the electrical resistivity measurements are given in Table V. The order of magnitude and the behavior with temperature suggest that Yb_2RuH_6 like $Eu_2RuH_6^3$ is also a semiconductor. A graph of the log ρ vs. T^{-1} is shown in Figure 3. The data fitted the equation $\rho = \rho_0 e^{-\Delta E/2kT}$. From the slope of the line, ΔE was found to be 0.18 eV.

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Registry No. Yb₂RuH₆, 60594-81-2; YbH₂, 13598-40-8.

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